इंटरनेट मानक

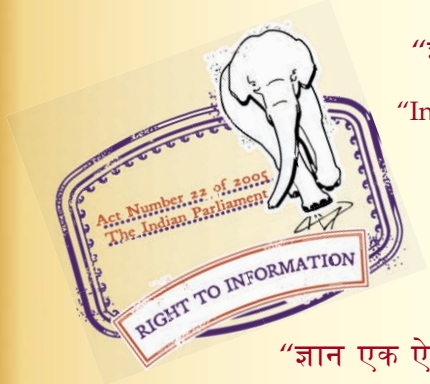
Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 12015 (1987): Cypermethrin Technical [FAD 1: Pesticides and Pesticides Residue Analysis]



"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

IS : 12015 - 1987
(Reaffirmed 2012)

Indian Standard

SPECIFICATION FOR CYPERMETHRIN TECHNICAL

UDC 632·951 CYP

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3

December 1987

Indian Standard

SPECIFICATION FOR CYPERMETHRIN TECHNICAL

Pest Control Sectional Committee, AFCDC 6

Chairman	*Representing*
DR R. L. RAJAK	Directorate of Plant Protection, Quarantine & Storage (Ministry of Agriculture & Rural Development), Faridabad
Members	
DR P. K. AGGARWAL	IDMA Laboratories, Chandigarh
SHRI ACHINT CHAUDHRY (*Alternate*)	
DR H. L. BAMI	Pesticides Residue Analysis Sectional Committee, AFDC 56, BIS
DR B. BANERJEE	Tea Research Association, Jorhat
DR G. SATYANARAYANA (*Alternate* I)	
SHRI S. C. DAS (*Alternate* II)	
DR S. R. BAROOAH	Pesticides Formulators Association of India (SSI), Bombay
SHRI O. K. BHATNAGAR	Agriculture Department, Government of Uttar Pradesh, Lucknow
SHRI RAM NARAIN SINGH (*Alternate*)	
SHRI N. S. BIRDIE	Shriram Institute for Industrial Research, Delhi
SHRI R. K. BANERJEE (*Alternate*)	
SHRI D. CHATTERJEE	Agriculture Department, Government of West Bengal, Calcutta
SHRI M. V. DESAI	Atul Products Limited, Atul
SHRI A. R. DESAI (*Alternate*)	
SHRI G. D. GOKHALE	Bombay Chemicals Private Limited, Bombay
SHRI P. M. JADHAV (*Alternate*)	
SHRI B. K. VIJAYA GOPAL	Agromore Limited, Bangalore
SHRI S. K. RAMAN (*Alternate*)	
DR R. C. GUPTA	Central Insecticides Laboratory, Directorate of Plant Protection, Quarantine & Storage (Ministry of Agriculture & Rural Development), Faridabad
SHRI S. K. GHOSH (*Alternate* I)	
SHRI R. S. SHARMA (*Alternate* II)	
DR S. S. GUPTE	Bayer (India) Limited, Bombay
DR R. N. JUKAR (*Alternate*)	
DR V. N. IYER	Coromandel Indag Products India (P) Limited, Madras
DR M. O. ALIAS (*Alternate*)	

(*Continued on page* 2)

(*Continued from page* 1)

Members	*Representing*
SHRI N. L. KALRA	National Institute of Communicable Diseases, Delhi
SHRI G. C. JOSHI (*Alternate*)	
DR R. L. KALRA	Punjab Agricultural University, Ludhiana
DR R. P. CHAWLA (*Alternate*)	
DR KALYAN SINGH	C. S. A. University of Agriculture & Technology, Kanpur
DR S. K. KASHYAP	National Institute of Occupational Health (ICMR), Ahmadabad
SHRI M. P. SHAH (*Alternate*)	
DR KENCHAIAH	Department of Agriculture, Government of Karnataka, Bangalore
SHRI JASWANT SINGH KHOKHAR	Agriculture Department, Government of Punjab, Chandigarh
SHRI S. G. KRISHNAN	National Organic Chemical Industries Limited, Bombay
DR J. S. VERMA (*Alternate*)	
SHRI S. K. MAJUMDAR	Central Food Technological Research Institute (CSIR), Mysore
SHRI N. MUTHU (*Alternate*)	
SHRI B. P. S. MANN	Pesticides Association of India, New Delhi
DR B. S. SHETTY (*Alternate* I)	
SHRI T. V. JAGADISAN (*Alternate* II)	
SHRI K. S. MEHTA	Bharat Pulverising Mills Private Limited, Bombay
SHRI S. CHATTERJEE (*Alternate*)	
DR A. L. MOOKERJEE	Cynamid India Limited, Bombay
DR A. P. SHINTRE (*Alternate*)	
DR S. K. MUKERJI	In personal capacity (*296, SFS Gulmohar Enclave, New Delhi 110049*)
SHRI NAWAB SINGH	National Malaria Eradication Programme, Delhi
CH KRISHNA RAO (*Alternate*)	
SHRI Y. A. PRADHAN	Rallis India Limited, Bombay
SHRI M. L. SHAH (*Alternate*)	
DR P. S. RAMANATHAN	Gharda Chemicals Private Limited, Thane
DR A. M. MALTE (*Alternate* I)	
SHRI D. G. UDAS (*Alternate* II)	
DR N. K. ROY	Agricultural Chemicals Division, Indian Agricultural Research Institute (ICAR), New Delhi
SHRI H. V. RUPAREL	Gujarat Insecticides Limited, Ankleshwar
DR J. K. ASTIK (*Alternate*)	
SHRI P. K. RAO	Ministry of Defence (DGI)
SHRI P. N. AGARWAL (*Alternate*)	
SECRETARY (CIB & RC)	Central Insecticides Cell, Directorate of Plant Protection, Quarantine & Storage (Ministry of Agriculture & Rural Development), Faridabad
SHRI V. C. BHARGAVA (*Alternate* I)	
DR SHANTI SARUP (*Alternate* II)	
DR K. D. SHARMA	Directorate General of Technical Development, New Delhi
SHRI N. R. NARAYANAN (*Alternate*)	
SHRI V. C. SHARMA	Department of Agriculture, Government of Rajasthan, Jaipur
DR K. N. SHRIVASTAVA	Shaw Wallace Company Limited, Calcutta
SHRI R. N. ROYCHOWDHURY (*Alternate*)	

(*Continued on page* 11)

Indian Standard

SPECIFICATION FOR CYPERMETHRIN TECHNICAL

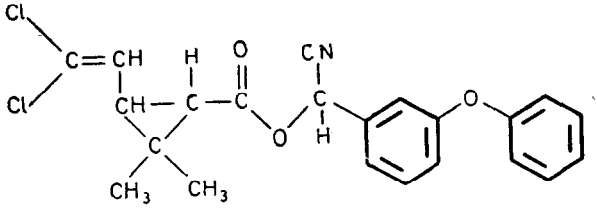
0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 30 June 1987, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Cypermethrin, technical, is an insecticide employed in the preparation of insecticidal formulations for the control of agricultural insect pests.

0.3 Cypermethrin is the common name accepted by International Organization for Standardization (ISO) for (*RS*) — α — Cyano — 3 — phenoxybenzyl (1, RS) — *cis trans* — 3 — (2, 2-dichlorovinyl) — 2, 2—dimethyl cyclopropane carboxylate. The empirical and structural formulae, and the molecular mass of cypermethrin are indicated below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{22}H_{19}Cl_2NO_3$		416·32



0.4 In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968, and Rules framed thereunder. However, this standard is subject to the restrictions imposed under the Act and Rules, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for cypermethrin, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a viscous liquid to semi solid mass. It shall be free from extraneous matter.

NOTE — The material shall be heated on a water-bath and homogenized before taking samples.

2.2 The material shall comply with the requirements specified in Table 1.

TABLE 1 REQUIREMENTS FOR CYPERMETHRIN, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Appendix of this Standard	Cl No. of IS : 6940-1982*
(1)	(2)	(3)	(4)	(5)
i)	Cypermethrin content, percent by mass, *Min*	92·0	A	—
ii)	Acidity (as H_2SO_4), percent by mass, *Max*	0·3	—	11.3.2
iii)	Moisture content, percent by mass, *Max*	0·2	—	4.1

*Methods of test for pesticides and their formulations (*first revision*).

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to the requirement given in IS : 8190 (Part 2)-1980*.

3.2 Marking — The containers shall bear legibly and indelibly the following information and other additional information as required under the *Insecticides Act* and Rules:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;
e) Cypermethrin content, percent (*m/m*);
f) Net mass of the content; and
g) The cautionary notice as worded in the *Insecticides Act* and Rules.

*Requirements for packing of pesticides: Part 2 Liquid pesticides (*first revision*).

3.2.1 Each container may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in IS : 10946-1984*.

5. TESTS

5.1 Tests shall be carried out by the methods referred to in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF CYPERMETHRIN CONTENT

A-0. GENERAL

A-0.1 Either of the three methods, namely, HPLC method (*see* **A-1**), GLC method (*see* **A-2**) or UV spectrophotometric method (*see* **A-3**) may be used for determination of cypermethrin content. HPLC and GLC methods will be the referee methods in case of dispute.

A-1. HPLC METHOD

A-1.1 Principle — A HPLC unit with a UV detector is used for this assay. Using solutions containing known amounts of standard cypermethrin and internal standard, the response factor, *RF*, for cypermethrin and internal standard approach is arrived at. A solution containing known mass of

*Methods of sampling for technical grade pesticides.

†Specification for water for general laboratory use (*second revision*).

the technical sample and internal standard is injected subsequently. The percentage of cypermethrin in the sample is then computed by standard relationship.

A-1.2 Apparatus

A-1.2.1 *High Performance Liquid Chromatograph* — Equipped with a printer-plotter-*cum*-integrator and UV detector. The suggestive parameters are given below and these operating conditions can be varied provided standardization is done:

Column	Stainless steel: 25 cm × 4·6 mm. Packed with silica of 5 μm particle size
Detector	UV (280 nm)
Solvent system	Carbon tetrachloride — 80 percent (*v/v*) Chloroform — 20 percent (*v/v*)
Solvent flow rate	0·4 ml/min
Pressure	1 500 p.s.i.
Chart speed	0·25 cm/min
Sample size	10 μl

A-1.2.2 *Volumetric Flask* — 100-ml capacity.

A-1.3 Reagents

A-1.3.1 *Internal Standard* — di-*n*-butyl phthalate, AR grade.

A-1.3.2 *Carbon Tetrachloride* — Spectroscopic grade.

A-1.3.3 *Chloroform* — Spectroscopic grade.

A-1.3.4 *Cypermethrin Standard* — of known purity.

A-1.4 Preparation of Standard and Calibration

A-1.4.1 Weigh accurately 1·0 g of internal standard in 100-ml volumetric flask and make up to volume using carbon tetrachloride — chloroform mixture [80 : 20 (*v/v*)]. This will give a 10 mg/ml solution of internal standard.

A-1.4.2 Weigh accurately 0·5 g of standard cypermethrin in 100-ml volumetric flask and make up volume using carbon tetrachloride— chloroform mixture. This will give 5 mg/ml of standard solution. Pipette out 4·0 ml of this solution in 50-ml volumetric flask. Add 5 ml of internal standard and mix well. Make up to mark with solvent system. Make similar standard solution but employing 6·0, 8·0, 10·0 and 12·0 ml of standard cypermethrin solution.

A-1.4.3 Introduce 10 μl of the five standard solution in HPLC unit. From the integrator, print out and note down the peak areas of the cypermethrin standard and internal standard peaks in all cases. Adjust the attenuation in such a way that cypermethrin standard and internal standard peaks are obtained within the scale in all the cases.

A-1.4.4 Draw a graph connecting the mass and area ratios. Measure the slope, *RF*, of the graph which will also be response factor for cypermethrin standard.

A-1.5 Procedure

A-1.5.1 Weigh 0·5 g of sample in 100-ml volumetric flask and make up volume with carbon tetrachloride—chloroform mixture. Pipette out 10 ml of this solution in 50-ml volumetric flask. Add 5 ml of internal standard. Mix well and make up the volume with solvent mixture.

A-1.5.2 Introduce 10 μl of this solution in HPLC. From the integrator print out and note down the peak areas of cypermethrin and internal standard. Calculate the cypermethrin content.

A-1.6 Calculation

$$\text{Cypermethrin content, percent by mass} = \frac{RF \times A_1 \times m_1}{A_2 \times m_2} \times 100$$

where

RF = response factor;

A_1 = area of cypermethrin peak in the sample;

m_1 = mass, in g, of the internal standard added;

A_2 = area of the internal standard peak; and

m_2 = mass of the cypermethrin sample taken for analysis.

A-2. GLC METHOD

A-2.1 Principle — A GLC unit with FID is used for this determination. Using solutions containing known amounts of the standard cypermethrin and internal standard, the response factor, *RF*, for cypermethrin and internal standard is arrived at. A solution containing a known mass of sample solution and internal standard is injected subsequently into GLC unit. The percentage of cypermethrin is then computed by standard relationship.

A-2.2 Apparatus

A-2.2.1 *Gas Liquid Chromatograph* — Equipped with flame ionization detector (FID) coupled to a printer-plotter-*cum*-integrator. The

suggestive operative parameters are given below but can be changed in any other equipment, provided standardization is done:

Column	75 cm × 3 mm id Stainless steel packed with 3 percent Dexil 300 on chromosorb. W-HP, 100-120 mesh.
Gas flow	
Carrier gas (N_2)	40 ml/min
Fuel gas (H_2)	40 ml/min
Air	300 ml/min
Temperature	
Column oven	240°C
Detector	270°C
Injection port	270°C

A-2.2.2 *Micro Syringe* — 10 µl capacity.

A-2.3 Reagents

A-2.3.1 *Toluene*

A-2.3.2 *Cypermethrin Standard* — of known purity.

A-2.3.3 *Internal Standard* — Dicyclohexyl phthalate.

A-2.4 Preparation of Standard and Calibration

A-2.4.1 Weigh accurately 5·0 g of internal standard in a 25-ml volumetric flask and make up to volume using toluene. This will contain 200 mg/ml of internal standard.

A-2.4.2 Weigh accurately 7·5 g of standard cypermethrin in 25-ml volumetric flask and make up to volume with toluene. Pipette 6·0 ml of this solution into another 25-ml volumetric flask. Add 5·0 g of internal standard solution (**A-2.4.1**) and make up to volume with toluene. Prepare similar mixtures of cypermethrin and internal standard containing 4·0, 5·0, 7·0 and 8·0 ml of standard cypermethrin solution containing 5·0 ml of internal standard. Make each to 25-ml with toluene.

A-2.4.3 Inject 2·0 µl of standard solution in GLC and from the integrator, print out and note down the peak areas of cypermethrin and internal standard in all the cases. Adjust the attenuation in such a way that cypermethrin and internal standard peaks are obtained within the scale in all the cases. Draw a graph between the mass and area ratio. Measure the slope, *RF*, of the graph which is also response factor for cypermethrin.

A-2.5 Procedure

A-2.5.1 Weigh accurately about 7·5 g of the sample in 25-ml volumetric flask and make up to volume with toluene. Pipette out 6·0 ml of this solution in another 25-ml volumetric flask, add 5 ml internal standard, mix well and make up the volume with toluene.

A-2.5.2 Inject 1·0 μl of the sample solution in GLC. From the integrator, print out and note down the peak areas of cypermethrin and internal standard peaks. Compute the percentage.

A-2.6 Calculation

$$\text{Cypermethrin content, percent by mass} = \frac{RF \times A_1 \times m_1}{A_2 \times m_2} \times 100$$

where

RF = response factor;

A_1 = area of the cypermethrin peak in the sample;

m_1 = mass, in g, of internal standard added;

A_2 = area of the internal standard peak; and

m_2 = mass, in g, of the sample taken for the test.

A-3. UV SPECTROPHOTOMETRIC METHOD

A-3.1 Principle — Absorbance of the sample solution in chloroform is measured at 279 nm against the solvent. The cypermethrin content of the sample is then computed by making use of a calibration graph prepared earlier.

A-3.2 Apparatus

A-3.2.1 *UV Spectrophotometer*

A-3.2.2 *Quartz Cell* — Matched pair with path length of 1·000 cm.

A-3.2.3 *Volumetric Flasks*

A-3.3 Reagents

A-3.3.1 *Chloroform* — Spectroscopic grade.

NOTE — The absorbance of this solvent shall not exceed 1·000, 0·300, 0·005, 0·005 and 0·005 at 245, 250, 275, 300 and 400 nm respectively. The spectral absorbance curve shall be smooth throughout this range and shall not show any extraneous impurity peaks.

A-3.4 Procedure

A-3.4.1 *Standard Preparation* — Weigh accurately 100 mg of standard cypermethrin sample in a 100-ml volumetric flask. Dissolve it in chloroform and make up to volume with chloroform. This solution will contain 1 000 μg/ml of cypermethrin. Pipette out 3·0, 4·0, 5·0 and 6·0 ml of solution in 50-ml volumetric flask and dilute to make with chloroform. This will give 60, 80, 100 and 120 μg/ml cypermethrin respectively.

A-3.4.2 *Solution Preparation* — Weigh out accurately 100 mg of the sample in clean and dry 100-ml volumetric flask and make up the volume with chloroform. Take 5·0 ml of this solution in another 50-ml volumetric flask and make up the volume with chloroform.

A-3.4.3 *Determination* — Fill the matched pair of cells with chloroform and measure cell error. It shall be less than 0·005 at 279 nm. Obtain the absorbance values at 279 nm of the four standard solutions in the cell against solvent blank. Draw a graph of absorbance values against cypermethrin concentration. Measure the slope of calibration graph. Measure the absorbance of the sample solution at 279 nm against solvent blank. Correct the absorbance value for the cell error, if any, and calculate the cypermethrin content of the sample.

A-3.5 Calculation

$$\text{Cypermethrin content, percent by mass} = \frac{E_s \times 100}{S \times m}$$

where

E_s = the final corrected absorbance of the sample;

S = slope of the calibration graph; and

m = mass, in mg, of the sample taken for the test.

(*Continued from page* 2)

Members	*Representing*
SHRI J. L. SIPAHIMALANI SHRI A. H. KARANCHI (*Alternate*)	CMA Laboratories, Bombay
DR R. SOMAN SHRI K. RATHI (*Alternate*)	Sudarshan Chemical Industries Limited, Pune
DR R. V. VENKATARATNAM DR NAGABHUSHAN RAO (*Alternate*)	Regional Research Laboratory (CSIR), Hyderabad
SHRI T. PURNANANDAM, Director (Agri & Food)	Director General, BIS (*Ex-officio Member*)

Secretary

SHRI LAJINDER SINGH
Joint Director (Agri & Food), BIS

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

AMENDMENT NO. 1 JANUARY 1989
TO
IS : 12015 - 1987 SPECIFICATION FOR CYPERMETHRIN, TECHNICAL

(*Page* 4, *Table* 1) — Insert the following after Sl No. (iii) under the respective columns:

(1)	(2)	(3)	(4)	(5)
iv)	3-phenoxybenzaldehyde (MPB) content, percent by mass, *Max*	2·0	B	—
v)	*Cis-trans* ratio, percent of nominal value	*Cis Min* 40·0 *trans Max* 60·0	C	—

(*Page* 10) — Insert the following appendices after **A-3.5**:

APPENDIX B

[*Table* 1, *Item* (iv)]

DETERMINATION OF 3-PHENOXYBENZALDEHYDE (MPB)

B-0. PRINCIPLE

B-0.1 A GLC unit with FI detector is used for this determination. Using solutions containing known amounts of the standard 3-phenoxybenzaldehyde (MPB), sample and the internal standard (*Is*), the response factor *R*, for MPB in the (*Is*) approach is arrived at. A solution containing known mass of the cypermethrin sample (under investigation) and *Is* is injected subsequently into the GLC unit. The percentage of MPB in the sample is then computed by the standard relationship.

B-1. APPARATUS

B-1.1 Gas Liquid Chromatograph — Suitable for analysis when operated under the following suggestive operating conditions. These conditions can be varied provided standardization is done.

Column	6′ × 1/4″ glass column filled with 3 percent. OV-225 coated on Chromosorb WHP (80-100 mesh)
Column temperature	210°C
Injection port	260°C
Detector	260°C
Carrier gas	Nitrogen at 40 ml/min

B-1.2 Volumetric Flask

B-1.3 Microsyringe

B-2. REAGENTS

B-2.1 Metaphenoxy Benzaldehyde — AR Grade.

B-2.2 Chloroform — AR Grade.

B-2.3 Di-*n*-Butyl Phthalate — Internal standard AR Grade.

B-3. PREPARATION OF STANDARDS AND CALIBRATION

B-3.1 Weigh out accurately 2·5 g of di-*n*-butyl phthalate into a 50-ml volumetric flask and make up to volume with chloroform. This will give a solution containing 50 mg/ml of the internal standard.

B-3.2 Weigh out accurately 2·5 g of standard metaphenoxy benzaldehyde into a 50-ml volumetric flask and make up to volume with chloroform. This will give a solution containing 50 mg/ml of metaphenoxy benzaldehyde. Pipette out 1·0, 2·0, 3·0, 4·0 and 5·0 ml of this metaphenoxy benzaldehyde solution into a clean dry 10-ml flask. Add 5 ml of *Is* solution in each flask. Make up the volume with chloroform in the first four cases.

B-3.3 Inject 0·2 μl of the standard solutions described under **B-3.2** into the GIC unit. From the integrator print out and note down the peak areas of the metaphenoxy benzaldehyde and internal standard peaks.

B-3.4 Procedure

B-3.4.1 Weigh out accurately 5·0 g of the cypermethrin sample into a 10-ml volumetric flask. Measure out 5 ml of the *Is* solutions and make up to the mark with chloroform.

B-3.4.2 Introduce 0·2 μl of this sample solution into GIC unit. From the integrator print out, note down the peak areas of metaphenoxy benzaldehyde and internal standard and internal standard peaks. Compute the percentage of the metaphenoxy benzaldehyde content in the sample.

B-3.5 Calculation

$$\text{3-phenoxy benzaldehyde content, percent by mass} = \frac{R \times A_1 \times m_1}{A_2 \times m_2} \times 100$$

where

R = response factor ;

A_1 = area of the 3-phenoxy benzaldehyde peak obtained while analyzing the sample;

m_1 = mass of the internal standard added;

A_2 = area of the internal standard peak; and

m_2 = mass of the sample taken for analysis.

APPENDIX C

[*Table* 1, *item* (v)]

DETERMINATION OF THE CIS-TRANS ISOMER RATIO IN CYPERMETHRIN, TECHNICAL

C-1. HPLC METHOD

C-1.1 Principle — The sample is subjected to HPLC analysis and the isomer distribution arrived at from the area of the peaks representing each isomer.

C-1.2 Apparatus

C-1.2.1 *High performance liquid chromatograph* — equipped with a printer plotter cum integrator and U.V. detector. The suggestive HPLC operating conditions are given below and they can be varied, depending upon instrument, provided standardization is done :

Column	Silica 5 μm 25 cm × 4·6 mm, stainless steel
Solvent system	1 percent (*v/v*) di-isopropyl ether in *n*-pentane
Detector	U.V. at 280 nm
Flow rate	0·6 ml/min
Pressure	980 psi
Sample size	10 μl

C-1.2.2 *Standard Volumetric Flasks*

C-1.3 Reagents

C-1.3.1 *Di-isopropyl Ether* — AR Grade.

C-1.3.2 *n-pentane* — AR Grade.

C-1.4 Procedure

C-1.4.1 Weigh out accurately 1·0 g of the cypermethrin, technical sample into a 100-ml volumetric flask and make up to volume using *n*-pentane containing 1 percent (*v/v*) di-isopropyl ether. Inject 10 μl of the solution into the HPCL equipment, and record the chromatogram. This will consist of four major peaks. This first two peaks represent the *cis-isomer*, while the rest two represent the *trans*-isomer. Calculate the total area of each of the two pairs of peaks from the data printed out by the printer plotter. Compute the *cis-trans* isomer ratio as indicated in C-1.5.

C-1.5 Calculation

$$\text{Percentage of } cis\text{-isomer in the cypermethrin, technical sample} = \frac{\text{Total area of the first two major peaks} \times 100}{\text{Total area of all the four major peaks}}$$

$$\text{Percentage of } trans\text{-isomer in the cypermethrin, technical sample} = \frac{\text{Total area of the third and fourth major peaks} \times 100}{\text{Total area of all the four major peaks}}$$

Printed at New India Printing Press, Khurja, Ind

AMENDMENT NO. 2 APRIL 1991

TO

IS 12015 : 1987 SPECIFICATION FOR CYPERMETHRIN, TECHNICAL

[*Page* 4, *Table* 1 (*see* also *Amendment No.* 1)] — Delete 'Sl No. (iv)' and entries under its respective columns, and renumber 'Sl No. (v)' as 'Sl No. (iv)'.

(*Pages* 1, 2 *and* 3 *of Amendment No.* 1) — Delete 'Appendix B' and redesignate 'Appendix C' as 'Appendix B'.

(FACD 1)

Printed at New India Printing Press, Khurja, India

AMENDMENT NO. 3 MARCH 1992
TO
IS 12015 : 1987 SPECIFICATION FOR CYPERMETHRIN, TECHNICAL

(*Pages* 6 *and* 7, *clauses* A-1.4 *to* A-1.6) — Substitute the following for the existing clauses:

'A-1.4 Preparation of Internal Standard, Reference Standard and Sample Solutions

A-1.4.1 Weigh accurately 1 g of internal standard in a 100-ml volumetric flask and make up the volume using carbon tetrachloride-chloroform mixture [solvent system — 80 : 20 (*v/v*)]. This will give a 10 mg/ml solution of internal standard.

A-1.4.2 Weigh accurately 0.5 g of cypermethrin standard in a 100-ml volumetric flask and make up the volume using solvent system. This will give 5 mg/ml of standard solution. Take out 10.0 ml of this solution into a 50-ml volumetric flask with the help of a pipette. Add 5 ml of internal standard solution and mix well. Make up the volume to the mark with solvent system. Call this as Solution A.

A-1.4.3 Weigh accurately 0.5 g of sample in a 100-ml volumetric flask and make up the volume with solvent system. Take out 10.0 ml of this solution with a pipette into a 50-ml volumetric flask. Add 5 ml of internal standard solution, mix well. Make up the volume to the mark with solvent system. Call this as Solution B.

A-1.5 Procedure

A-1.5.1 Introduce 10 µl of Solutions A and B simultaneously into the HPLC unit. From the integrator, print out and note down peak areas of cypermethrin and internal standard in both cases. Calculate the cypermethrin content.

A-1.6 Calculation

Cypermethrin content,

$$\text{percent by mass} = \frac{m_1 \times A_2 \times A_3}{m_2 \times A_1 \times A_4} \times P$$

where

m_1 = mass, in g, of cypermethrin standard in Solution A;
A_2 = peak area of cypermethrin in Solution B;
A_3 = peak area of internal standard in Solution A;
m_2 = mass, in g, of cypermethrin in sample in Solution;
A_1 = peak area of cypermethrin standard in Solution A;
A_4 = peak area of internal standard in Solution B ; and
P = percent purity of cypermethrin standard.'

(*Pages* 7, 8 *and* 9, *clauses* **A-2.2.1, A-2.4** *to* **A-2.6**) — Substitute the following for the existing clauses:

'**A-2.2.1** *Gas Liquid Chromatograph (GLC)* — Equipped with flame ionization detector (FID) coupled to a printer-plotter-cum-integrator. The suggestive operative parameters are given below but can be changed in any other equipment, provided standardization is done:

Column	90 cm × 3 mm id Stainless steel packed with 3 percent Dexil 300 or suitable equivalent on chromosorb. W-HP, 100-120 mesh.
Gas flow	
Carrier gas (N_2)	40 ml/min
Fuel gas (H_2)	40 ml/min
Air	300 ml/min
Temperature	
Column oven	240°C
Detector	270°C
Injection port	270°C

A-2.4 Preparation of Internal Standard, Reference Standard and Sample Solutions

A-2.4.1 Weigh accurately 2.0 g of internal standard in a 100-ml volumetric flask and make up the volume using toluene. This will contain 20 mg/ml of internal standard.

A-2.4.2 Weigh accurately 375 mg of cypermethrin standard in a 25-ml volumetric flask and make up the volume with toluene. Take out 10.0 ml of this solution with a pipette into another 25-ml volumetric flask, add 5.0 ml internal standard solution, make up the volume with toluene and mix well. Call this as Solution A. The solution will contain 6 mh/ml of cypermethrin.

A-2.4.3 Weigh accurately a quantity of sample containing equivalent to 375 mg of cypermethrin in a 25-ml volumetric flask and make up the volume with toluene. Take out 10.0 ml of this solution with a pipette into another 25-ml volumetric flask, add 5.0 ml of internal standard solution, make up the volume with toluene and mix well. Call this as Solution B. This solution will contain 6 mg/ml of cypermethrin.

A-2.5 Procedure

A-2.5.1 Inject 1.0 µl of Solutions A and B simultaneously into the GLC unit and from the integrator, print out and note down peak areas of the internal standard, cypermethrin standard and the sample.

A-2.6 Calculation

Cypermethrin content,

$$\text{percent by mass} = \frac{M_1 \times A_2 \times A_3}{M_2 \times A_1 \times A_4} \times P$$

where

M_1 = mass, in g, of cypermethrin standard in Solution A;
A_2 = peak area of cypermethrin in Solution B;
A_3 = peak area of internal standard in Solution A;
M_2 = mass, in g ,of cypermethrin in sample in Solution B;
A_1 = peak area of cypermethrin standard in Solution A;
A_4 = peak area of internal standard in Solution B; and
P = percent purity of cypermethrin standard.'

(*Pages* 9 *and* 10, *clauses* **A-3.4.1** *to* **A-3.5**) — Substitute the following for the existing clauses:

'**A-3.4.1** *Standard Preparation* — Weigh accurately 100 mg of standard cypermethrin in a 100-ml volumetric flask. Dissolve in chloroform and make up the volume to the mark. This solution will contain 1 000 µg/ml of cypermethrin.

Take 5.0 ml of this solution into a 50-ml volumetric flask and dilute it to the mark with chloroform. The final solution will contain 100 µg/ml of cypermethrin.

A-3.4.2 *Solution Preparation* — Weigh out accurately 100 mg of the sample in clean and dry 100-ml volumetric flask and make up the volume with chloroform. Take 5.0 ml of this solution in another 50-ml volumetric flask and make up the volume with chloroform. The final solution will contain 100 µg/ml of cypermethrin.

A-3.4.3 *Determination* — Fill the matched pair of cells with chloroform and measure cell error. It shall be less than 0.005 at 279 nm. Obtain the absorbance values at 279 nm of the standard and sample solutions in the cell against solvent blank. Correct the absorbance values for the cell error, if any, and calculate the cypermethrin content of the sample.

A-3.5 Calculation

$$\text{Cypermethrin content, percent by mass} = \frac{M_1 \times A_2}{M_2 \times A_1} \times P$$

where

M_1 = mass, in g, of the cypermethrin standard;

A_2 = absorbance of the sample;

P = percent purity of the cypermethrin standard;

M_2 = mass, in g, of the sample; and

A_1 = absorbance of the standard.'

(FAD 1)

Printed at New India Printing Press, Khurja, India